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THE EFFECTS OF PULSE PLATING ON LOW CONTRACTION CHROMIUM ELECTRODEPOSITS

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The pulse plating of low contraction (LC) chromium using low pulse frequencies (less than 50 Hz) and high pulse frequencies (greater than 90 Hz) was evaluated and compared to direct current (dc)-plated LC chromium with respect to microstructure and mechanical properties. Low frequency pulse plating significantly increases the hardness and cathode current efficiency (CCE) over dc-plated LC electrodeposits but does not reduce deposit stress. Hardness values in excess of 1175 KHN (50 g load) can be obtained with pulsing frequencies less than
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20. ABSTRACT (CONT'D)

26 Hz and duty cycles (percent on-time) less than 21 percent. CCE in excess of 22 percent can be obtained when pulsing frequencies are less than 12 Hz and duty cycles are less than 33 percent. This corresponds to a 54 percent increase in hardness and a 100 percent increase in CCE compared to dc-plated LC chromium. The maximum tensile strength obtained by low frequency pulse plating was 31 percent lower than that obtained by dc plating. High frequency pulse plating produces poor quality deposits with lower hardness, CCE, and ultimate tensile strength than that obtained with dc plating.

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INTRODUCTION

The electroplating of low contraction (LC) chromium resulted in chromium deposits which were crack-free and of relatively high tensile strength (ref 1). Further improvements by Pan, Miller, and Nelson (ref 2) on the LC chromium plating procedure have resulted in optimal direct current (dc)-plated deposits with an ultimate tensile strength (UTS) of 87,000 psi, hardness of 760 KHN (50 g load), and cathode current efficiency (CCE) of 11.9 percent. To further improve the quality of the LC chromium electrodeposits, the application of pulse plating on LC chromium was evaluated.

Pulse plating is a method of depositing metal on a substrate using interrupted direct current. The pulses, often employed at a rate of 10 to 10,000 times per second, have benefits which include increasing plating speeds, improving distribution and lowering deposit stress, refining grain structure, increasing ductility, increasing CCE, and reducing hydrogen embrittlement (ref 3). When the current is turned on in conventional dc plating, the metal ions near the cathode deposit immediately. The rate at which the metal ions deposit is greater than the diffusion rate of the ions, resulting in a concentration gradient in the immediate area of the cathode (ref 4). In addition, an electric field in the electrolyte aligns itself into a direction causing polarization near the edges of the cathode resulting in a thicker deposit near the end of the cathode (ref 5). When pulse current is used instead of conventional direct current, the current that is on for a short time is sufficient to deposit all the metal ions in the immediate vicinity of the cathode. During the off-time of the pulse cycle, solution equilibrium is re-established, eliminating any concentration gradient that would exist. In addition, the polarization level on the cathode surface is no longer concentrated near the end of the cathode but is evenly distributed across the layer of diffusion (ref 5).

Pulse plating affects only a small number of solutions (ref 6). This is because the diffusion rate of the metal ions not only varies from metal to metal, but it also depends upon the solution composition, temperature, pH, presence of additives, etc. Reports on the effects of pulse plating on chromium electrodeposition have been contradictory. Chin and Zhang (ref 7) reported that while pulse current increased coulombic efficiencies at some duty cycles, it tended to increase the internal stress of the chromium deposits resulting in an increase in cracks. On the other hand, Pearson and Dennis (ref 8) reported that pulse current reduced the internal stress and reduced or eliminated cracking of the chromium deposits.

This study systematically investigates the on/off pulse current (called ideal or unipolar pulse, as shown in Figure 1) of LC chromium to determine which pulsing conditions, if any, improve the mechanical and microstructural properties of dc-plated LC chromium electrodeposits. The purpose of this study is to determine the optimal on/off pulsing cycle by correlating it with the microstructure, topography, UTS, and hardness of the chromium deposit, and the CCE of the plating process.

EXPERIMENTAL PROCEDURE

The experimental procedure was carried out exactly as described in a previous report (ref 2) except for the electrodeposition process. A standard plating condition consisting of a peak current density of 100 A/dm^2, a chromic acid/sulfuric acid ratio of 100/1, and a chromium (Cr)(III) concentration of 4.0 g/l was used. A schematic diagram showing the pulse plating cell is shown in Figure 2.

In order to use the plated specimens in tensile tests, they were electroformed in the shape of cylinders. A copper tube with a 0.40-cm outer diameter

was used as the mandrel (cathode) and masked to a plating area of 5 cm^2. The cathode was placed vertically in the center of the beaker and rotated at 150 rpm during plating. A cylindrical mesh platinum-coated titanium anode, with a 10.8-cm diameter and a 12.7-cm length, was placed inside the beaker. The distance between the cathode and the anode was 5.2 cm. For x-ray diffraction (XRD) analysis, specimens were prepared by depositing pulse-plated LC chromium on a 2.8- by 2.8-cm copper cathode, masked on one side, and positioned and rotated in the same manner as the cylindrical cathode.

A rapid pulse power supply was used to pulse plate the LC chromium electrodeposits. A rectangular pulsating current, which fluctuated between zero and constant peak cathodic current value, was used. The waveform of the pulsed current was checked with an oscilloscope (Nicolet Model No. 2090). The plating time was adjusted so that the total charge transfer (A-hrs/dm^2) was kept constant at 240 A-hrs/dm^2.

The on/off pulsing was done in two parts: high frequency (short pulse cycle) and low frequency (long pulse cycle). For high frequency pulse plating, on-times of 0.1, 0.2, 0.4, 0.8, 1.0, and 10.0 ms were used, while off-times of 0.1, 0.2, 0.4, 0.8, and 1.0 ms were evaluated (see Figure 4). The frequency range of the pulsing cycle varied from 91 to 5000 Hz, while the duty cycle varied from 9 to 99 percent.

For low frequency pulse plating, on-times of 10, 20, 40, 80, and 100 ms were used, while off-times of 10, 20, 40, 80, and 100 ms were evaluated for each on-time (see Figure 8). The frequency range of the pulsing cycle varied from 5 to 50 Hz, while the duty cycle varied from 9.1 to 91 percent.

RESULTS AND DISCUSSION

Four specimens were prepared for each of the plating conditions investigated. The CCE, microhardness, and UTS results are the average of measurements for the four specimens.

Effects of High Frequency Pulse Plating

The quality, mechanical properties, and microstructure of the LC chromium deposits were greatly affected by the duty cycle and pulsing frequency. Pulsing in the short pulse regime (less than 10 ms) produced electrodeposits which were of poor quality. Most deposits, particularly those plated at on- and off-times less than 1.0 ms (pulsing frequencies greater than 900 Hz), contained large needlelike grains, poor adhesion (the deposit often flaking off the substrate), and increased stress.

The reason for such poor quality deposits at short pulsing times was explained by Saiddington (ref 9). After the first interruption, numerous growth centers corresponding to established nucleation sites develop. As the deposit grows thicker, growth centers coalesce and develop into larger, individual dome-like nodules. Saiddington clearly shows that as the number of interruptions increases, a growth of well-defined nodules develops. However, as the number of interruptions is increased beyond a critical point (a plating frequency greater than 900 Hz in our case), the surface topography becomes an incoherent conglomerate of high peaks and deep cavities. A high frequency of interruptions prevents the establishment of an oriented growth of the deposit and inhibits the development of well-defined growth centers that would eventually lead to the formation of nodules. When interruptions are spaced too closely, one nucleation is followed by another leading to a completely disoriented surface characterized by a dark gray appearance.

The effects of high frequency pulse plating on the hardness and CCE of LC chromium electrodeposits are shown in Figures 3a and 3b, and the corresponding frequency and duty cycle for each pulse cycle tested are shown in Figure 4. Figure 3a shows that while an individual on-time was held constant and the off-time systematically varied from 0.1 to 1.0 ms, nearly every high frequency cycle evaluated resulted in hardness values lower than the value of 760 KHN (50 g load) obtained under optimal dc-plating conditions (ref 2). At most off-times, hardness values tended to increase as the on-time increased suggesting that lower frequencies and higher duty cycles result in increased hardness.

Because there were a few exceptions to this trend, specific correlations were difficult to make. For example, at an off-time of 0.8 ms, an on-time of 0.1 ms (1111 Hz) resulted in a greater hardness than an off-time of 1.0 ms (714 Hz). These few exceptions agree with the results of Pearson and Dennis (ref 8) who showed that an increase in plating frequency resulted in an increase in hardness. Also of interest is the fact that hardness values at an on-time of 10.0 ms remain basically unchanged as the off-time is increased from 0.1 to 1.0 ms. This is strong evidence that the hardness is frequency and duty cycle-sensitive, since Figure 4 shows that the frequency and duty cycle change very little at an on-time of 10.0 ms.

The effects of high frequency pulse plating on the CCE are shown in Figure 3b. The highest CCE obtained, 16.5 percent, was only a slight improvement over the CCE obtained through dc plating of LC chromium. Many of the cycles tested, particularly those whose pulsing frequency was greater than 1000 Hz, resulted in lower CCE than that obtained with dc plating. This finding agrees with Han and Kwon (ref 10) who also reported that pulse plating at very high frequencies resulted in lower CCE than that obtained through dc plating. The highest CCE,

16.5 percent, occurring at 1.0 ms on-time and 0.4 ms off-time, corresponded to a 714 Hz pulsing frequency and a 71.4 percent duty cycle. Figure 3b suggests that for on-times less than 10 ms, the CCE can be increased by lowering the pulsing frequency, but the maximum CCE that can be expected is 16.5 percent.

Electrodeposits made by pulsing with high frequency (greater than 90 Hz) were of such poor quality, poor adhesion, and high stress, that UTS measurements were not possible.

The topographical photomicrographs of LC chromium pulse-plated with high pulse frequencies are shown in Figure 5. The morphology of LC chromium deposits plated with pulse current differed significantly from that obtained with direct current. The grain growth in the high frequency regime resembled isolated needlelike structures which became finer and darker in appearance as the pulsing frequency increased. Adhesion was very poor at frequencies greater than 2000 Hz (regardless of the duty cycle) and at frequencies around 100 Hz when the duty cycle was greater than 98 percent. Cracks were present in most of the coatings prepared under these test conditions.

Scanning electron microscope (SEM) photomicrographs showing the cross-sectional microstructure of pulse-plated LC chromium are shown in Figure 6. Only at pulsing frequencies near 100 Hz (on-times near 10 ms) did the microstructure represent a typical fibrous grain associated with LC chromium.

Effects of Low Frequency Pulse Plating

The effects of low frequency pulse plating on hardness, CCE, and UTS of LC chromium electrodeposits are shown in Figures 7a through 7c, and the corresponding frequency and duty cycle for each pulse cycle are shown in Figure 8. Figure 7b shows that a large fraction of the low frequency tests resulted in hardness values greater than those obtained at the optimal dc-plating conditions

(760 KHN). For on-times 40 ms or higher, an off-time of 10 ms consistently yielded the highest hardness values. For all conditions evaluated, an on-time of 10 ms consistently yielded the hardest deposits.

Maximum hardness values greater than 1175 KHN represent a 54 percent increase over the hardness values obtained with dc plating. These maximum hardness values occur when the on-time is 10 ms and the off-time is 40 ms or greater. In general, this suggests that hardness values in excess of 1175 KHN can be obtained by using pulsing frequencies at 25 Hz or less and duty cycles 20 percent or less (on-times not to exceed one-quarter of the off-time). A few exceptions to this conclusion did exist. For example, an on/off-time of 20/100 ms (8.3 Hz, 17 percent duty cycle) resulted in a hardness of only 880 KHN.

The effects of low frequency pulse plating on the CCE of LC chromium electrodeposits are shown in Figure 7c. It is quite apparent that low frequency pulse plating significantly improves the CCE over that obtained with dc plating. All conditions tested resulted in a CCE greater than 11.9 percent, which is the optimal CCE obtained by dc plating of LC chromium. The increase in CCE is a result of the decrease in concentration polarization and the elimination of the concentration gradient that occurs during dc plating.

As can be seen in Figure 7c, for each on-time tested, the CCE increased as the off-time increased, with each on-time reaching its largest CCE at an off-time of 100 ms. This suggests that lower frequencies and lower duty cycles result in CCE increases. A maximum CCE of 28 percent occurred at 40 ms on-time/100 ms off-time, and several values in excess of 25 percent were obtained. This 28 percent CCE is a 135 percent improvement over the CCE obtained by dc plating LC chromium. It can be concluded from Figure 7c that a CCE in excess of 22 percent can be obtained when the pulsing frequency is less

than 12 Hz and the duty cycle is less than 33 percent. This increase in CCE as the pulsing frequency decreased is in agreement with Han and Kwon (ref 10) who reported that pulse plating with pulsing frequencies less than 100 Hz resulted in a CCE 40 percent greater than that obtained from dc plating.

The effects of low frequency pulse plating on the UTS of LC chromium electrodeposits are shown in Figure 7a. The data from this study show that low frequency pulse-plated LC chromium electrodeposits were more stressed than those obtained through dc plating. This finding agrees with Chin and Zhang (ref 7) who reported that pulse current with on- and off-times less than 1000 ms increased the internal stress of the chromium deposits. Several of the deposits, including all of those plated at 10 and 20 ms on-time, were of such high stress that UTS test measurements were not possible.

None of the conditions yielded a UTS as high as 87,000 psi--the optimal tensile strength of dc-plated LC chromium. A maximum UTS of 60,000 psi (a 31 percent decrease over the UTS obtained with dc plating) was produced at 100 ms on-time/40 ms off-time. This was the only condition to yield a UTS greater than 40,000 psi. A frequency of 7.1 Hz and a duty cycle of 71 percent corresponded to this plating condition.

The topographical photomicrographs of LC chromium pulse-plated at low pulse frequencies are shown in Figure 9. For 10 and 20 ms on-times (pulsing frequencies between 50 and 33 Hz), the topography resembled a pattern of interwoven platelets, some with visible cracks such as the cracks shown for the 20 ms on-time/10 ms off-time sample. This structure would explain why UTS test measurements at 10 and 20 ms on-time were not possible. When the on-time was 40 ms or greater and the off-time 10 ms or greater, the topography resembled the hemispherical nodular appearance typical of dc-plated LC chromium.

SEM photomicrographs showing the cross-sectional microstructure of low frequency pulse-plated LC chromium are shown in Figure 10. As with the topography, the microstructure at on-times of 20 and 10 ms was of poor quality with a porous grain structure and cracks prevalent throughout. Only when the on-time was increased beyond 20 ms (pulsing frequencies less than 30 Hz and duty cycles greater than 70 percent) did the microstructure represent a typical fibrous grain associated with LC chromium.

A series of XRD patterns of low frequency pulse-plated LC chromium is shown in Figure 11. Two general trends can be observed from these patterns. First, as the off-time decreases (frequency increases), the crystal orientation becomes less random as evident by the intensity of the <110> peak decreasing. Second, as the on-time increases (frequency decreases), the relative intensity of the <211> peak increases suggesting a <211> preferred orientation at high on-times.

CONCLUSIONS

The pulse plating of LC chromium at high pulse frequencies (pulsing frequencies between 91 and 5000 Hz with duty cycles between 9 and 99 percent) and low pulse frequencies (pulsing frequencies between 5 and 50 Hz with duty cycles between 9 and 91 percent) has been evaluated and compared to dc-plated LC chromium. Based on the results of our experimental studies, the following conclusions can be made:

1. LC pulse-plated chromium using pulsing frequencies less than 50 Hz results in significant improvements in the CCE and increases in the hardness of the electrodeposits as compared with values obtained through dc plating.

2. Hardness values in excess of 1175 KHN can be obtained by pulse plating with pulsing frequencies at 25 Hz or less and duty cycles 20 percent or less.

This is a 54 percent increase in the optimal hardness obtained through dc plating of the LC chromium deposits.

3. CCE in excess of 22 percent can be obtained when pulsing frequencies less than 12 Hz and duty cycles less than 33 percent are used. This is a 100 percent increase over the optimal CCE obtained through dc plating.

4. Pulse plating using low pulse frequencies (less than 50 Hz) does not reduce the stress of the electrodeposits. Maximum tensile strength was actually 31 percent lower than that obtained through dc plating.

5. Pulse plating using high pulse frequencies (between 91 and 5000 Hz) results in poor quality deposits with poor adhesion, reduced hardness, CCE, UTS, and a needlelike grain structure.

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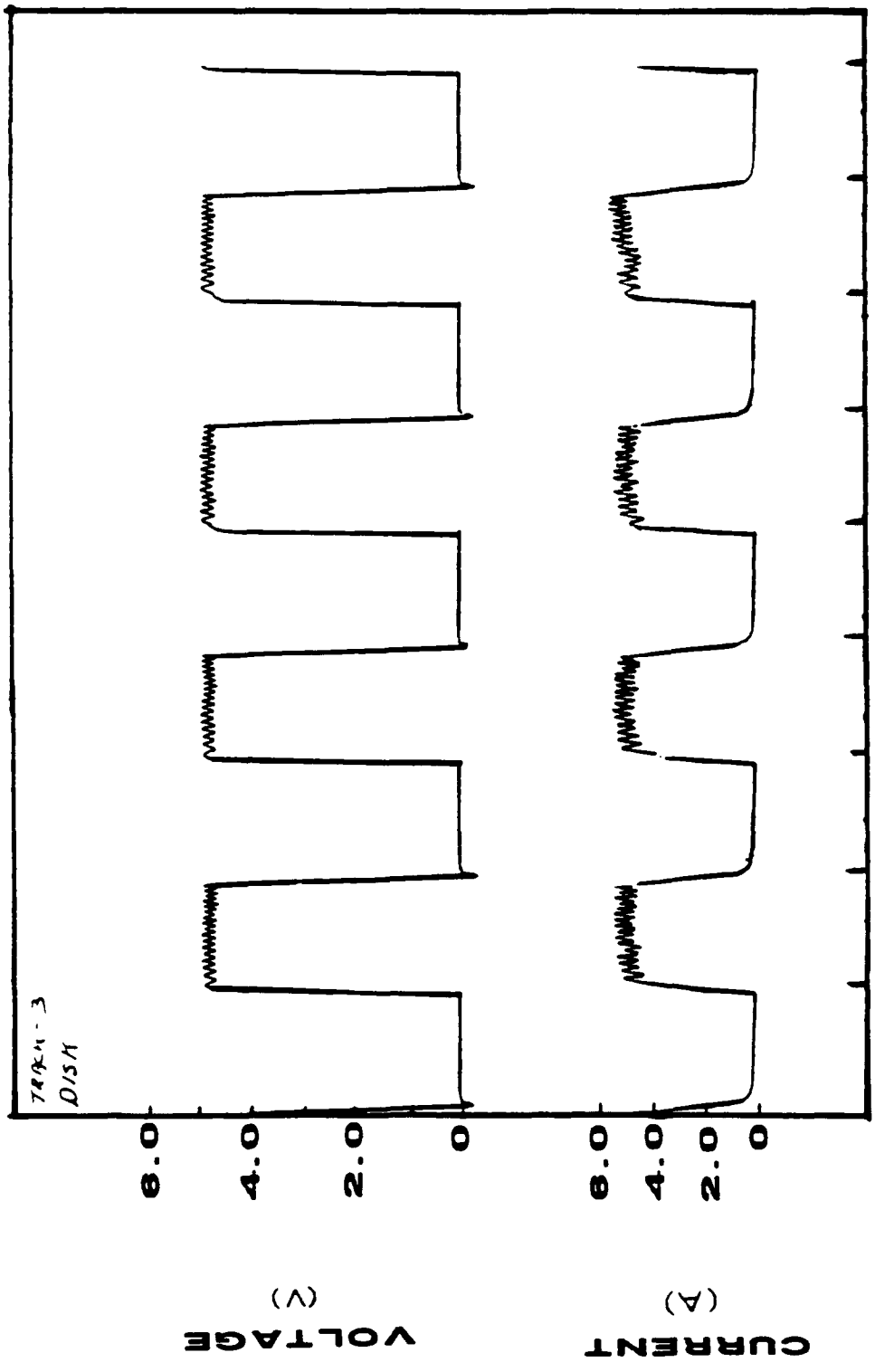

Figure 1. The voltage and current profile of high frequency pulse plating at 1.0 ms on-time/1.0 ms off-time.

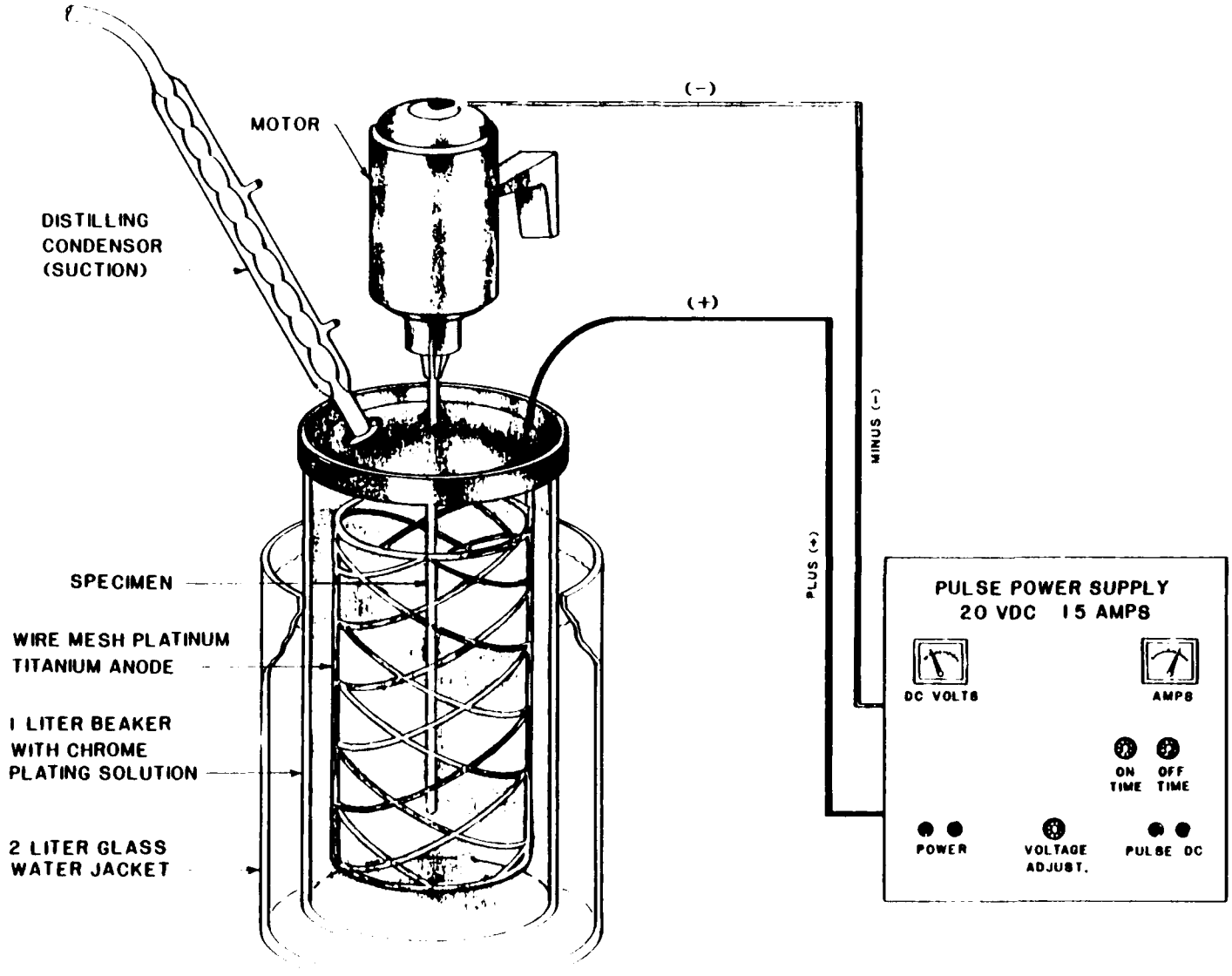

Figure 2. Schematic diagram of a pulse plating cell used to electrodeposit LC chromium.

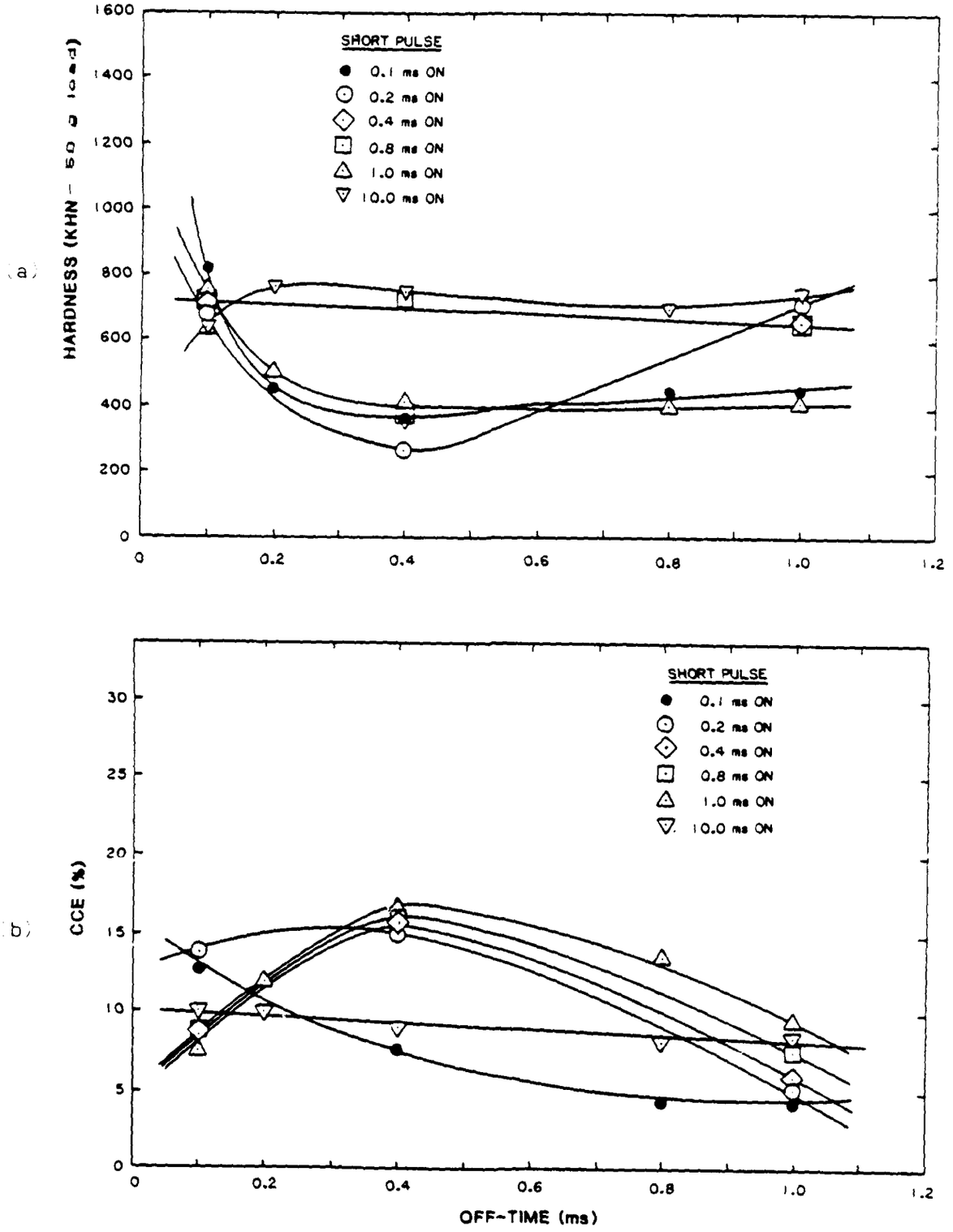

Figure 3. The effects of high frequency pulse plating on the (a) hardness and (b) CCE of LC chromium.

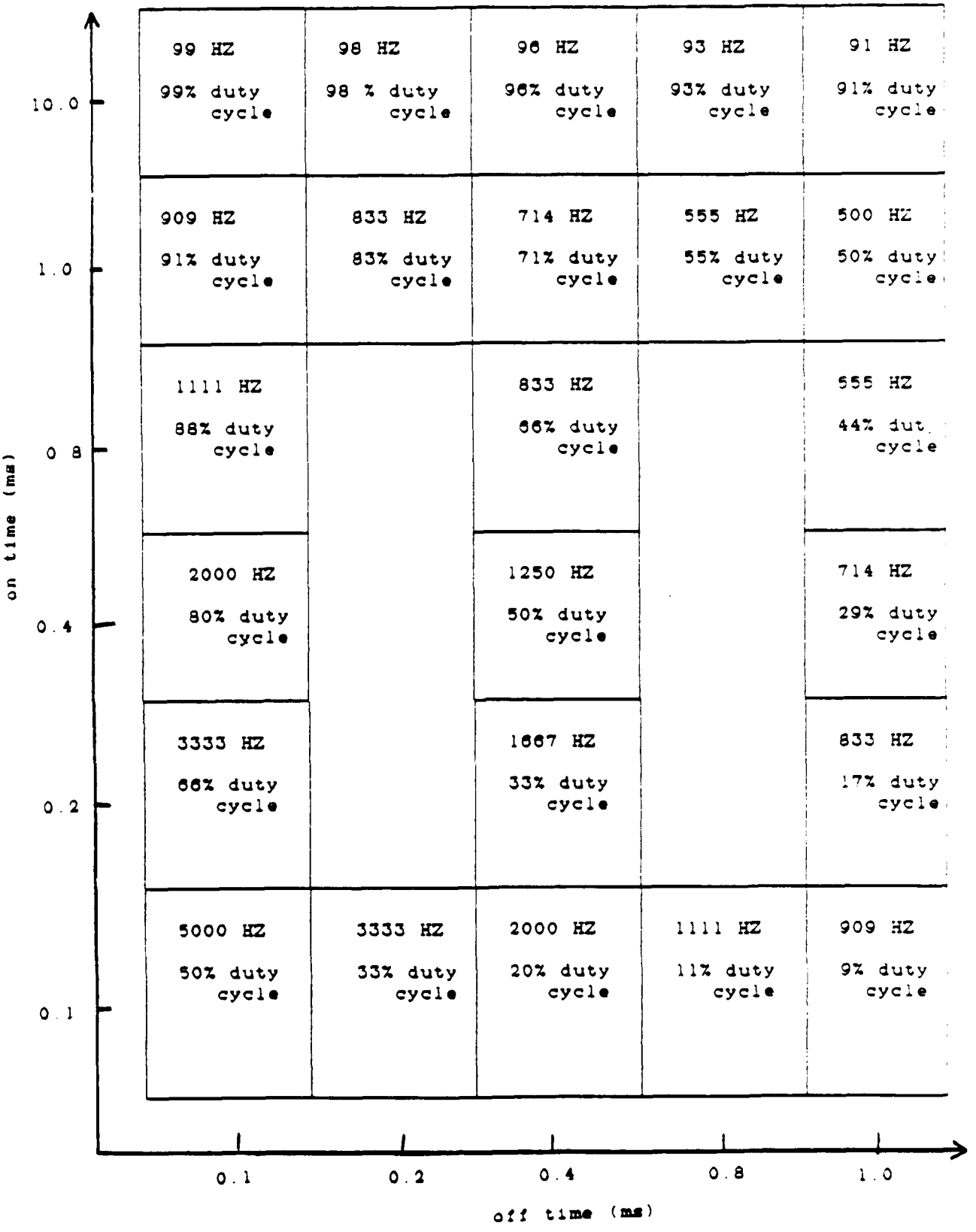

Figure 4. The frequency and duty cycle for each on/off-time condition evaluated during high frequency pulse plating.

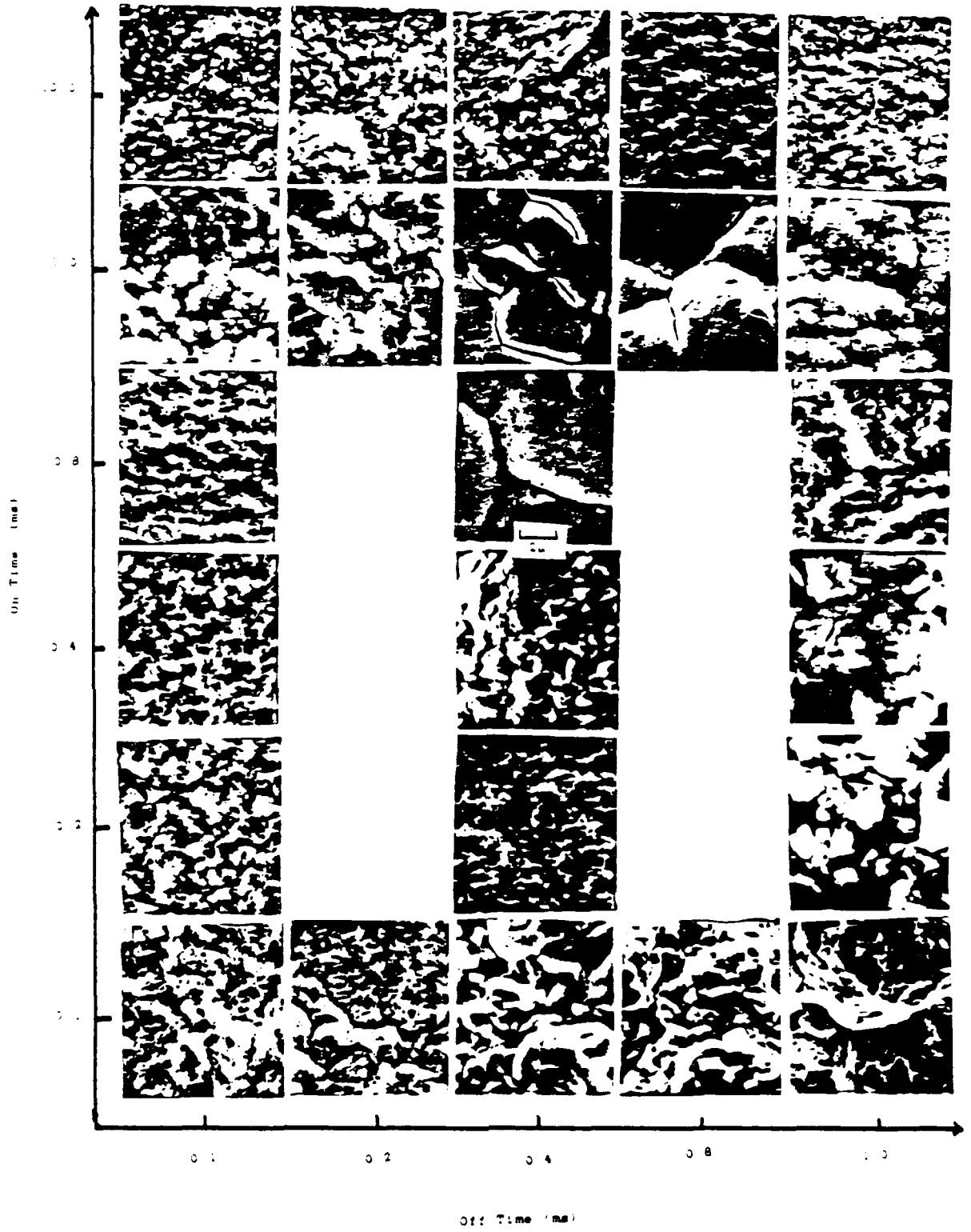

Figure 5. Topographical photomicrographs of LC chromium electrodeposited using high frequency pulse plating.

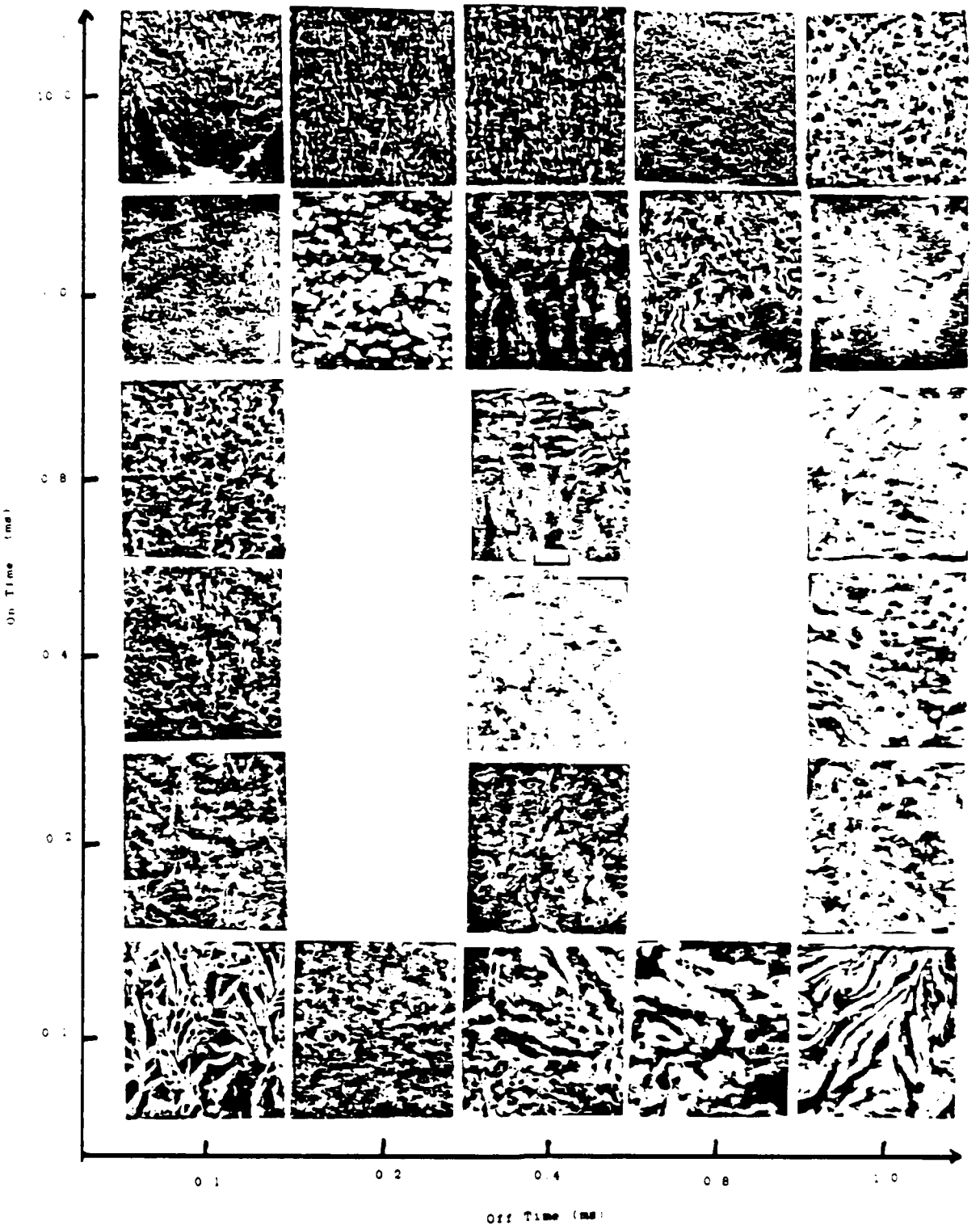

Figure 6. SEM photomicrographs of the cross-sectional microstructure of LC chromium electrodeposited using high frequency pulse plating.

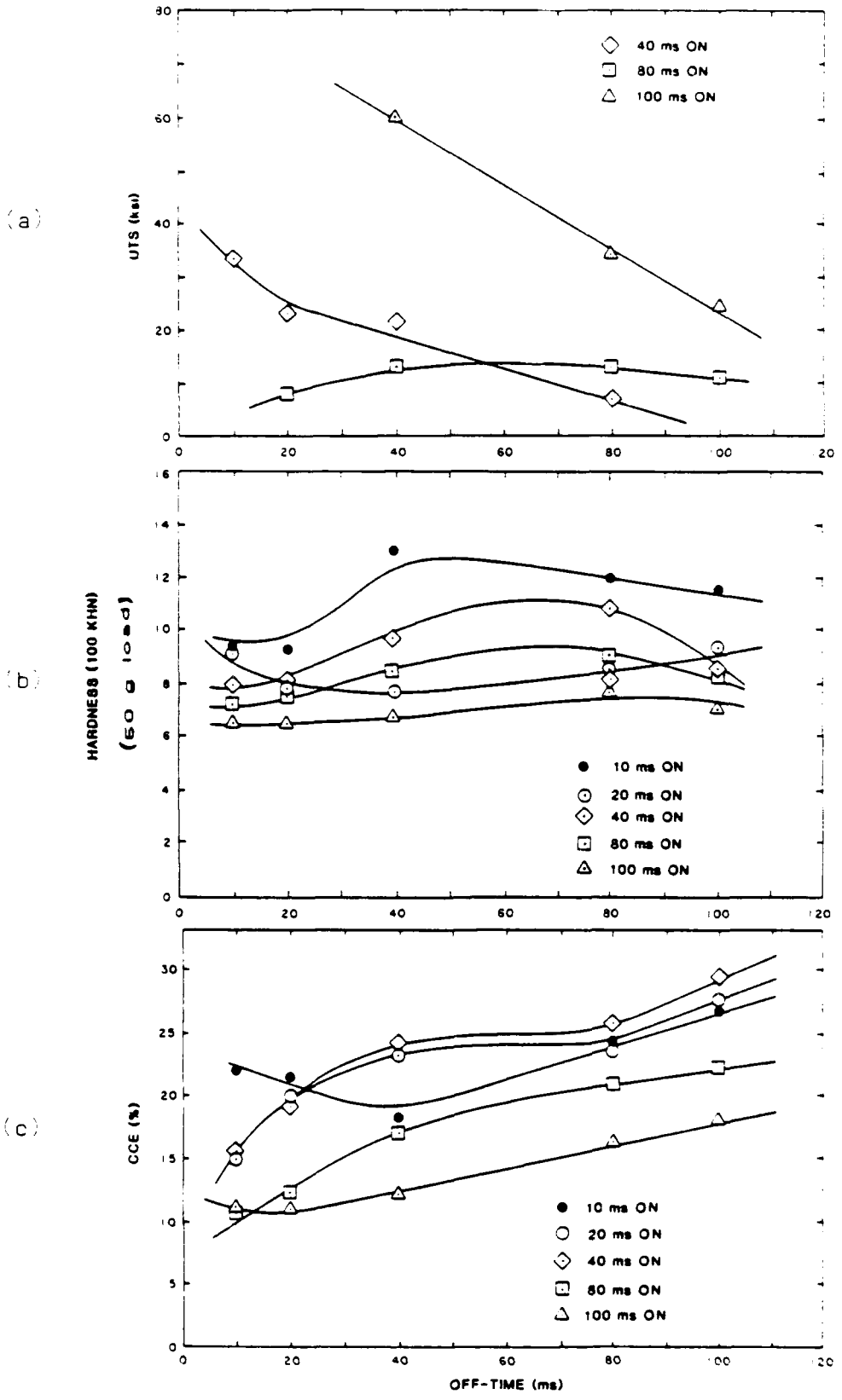

Figure 7. The effects of low frequency pulse plating on the (a) UTS, (b) hardness, and (c) CCE of LC chromium.

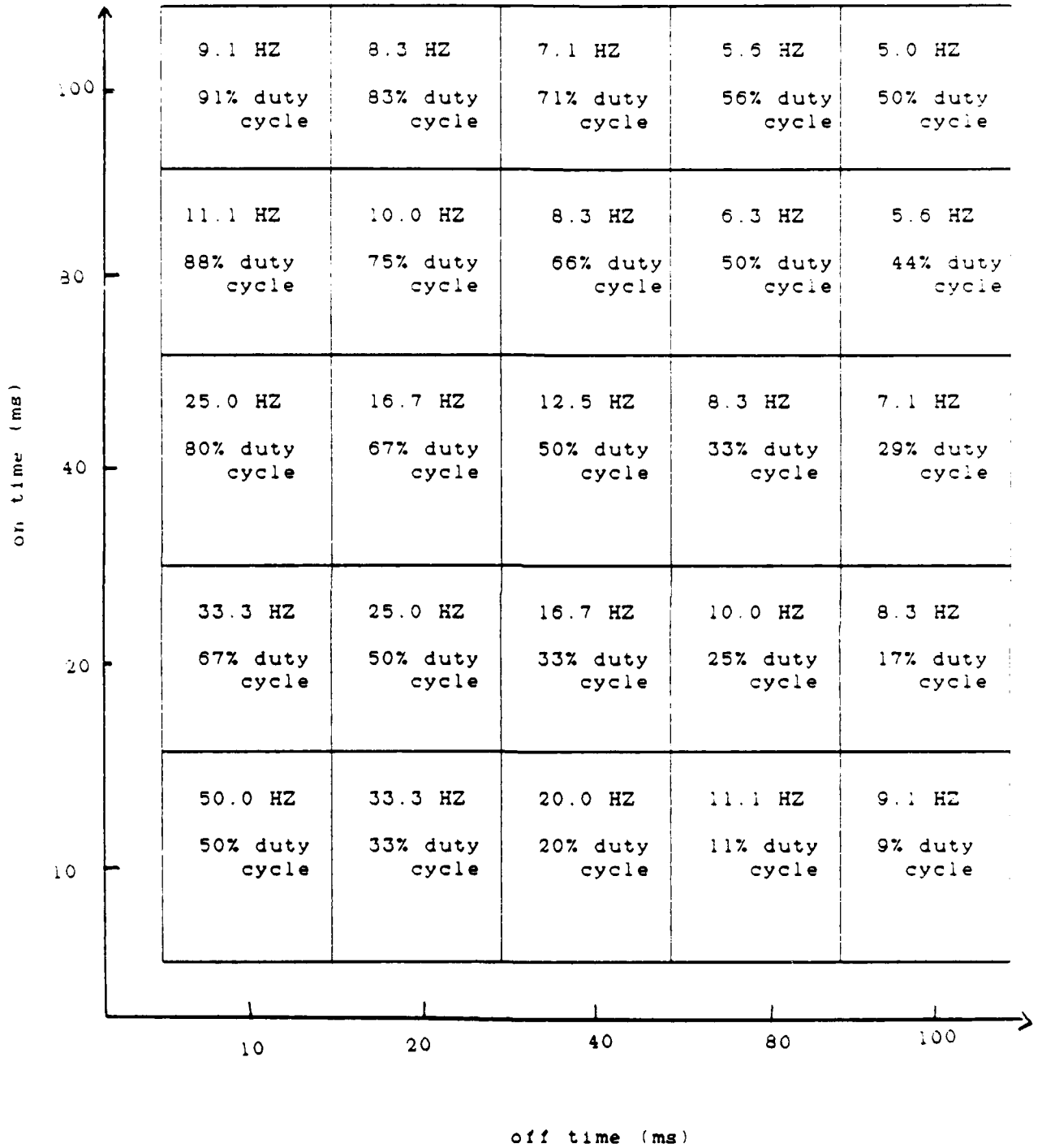

Figure 8. The frequency and duty cycle for each on/off-time condition evaluated during low frequency pulse plating.

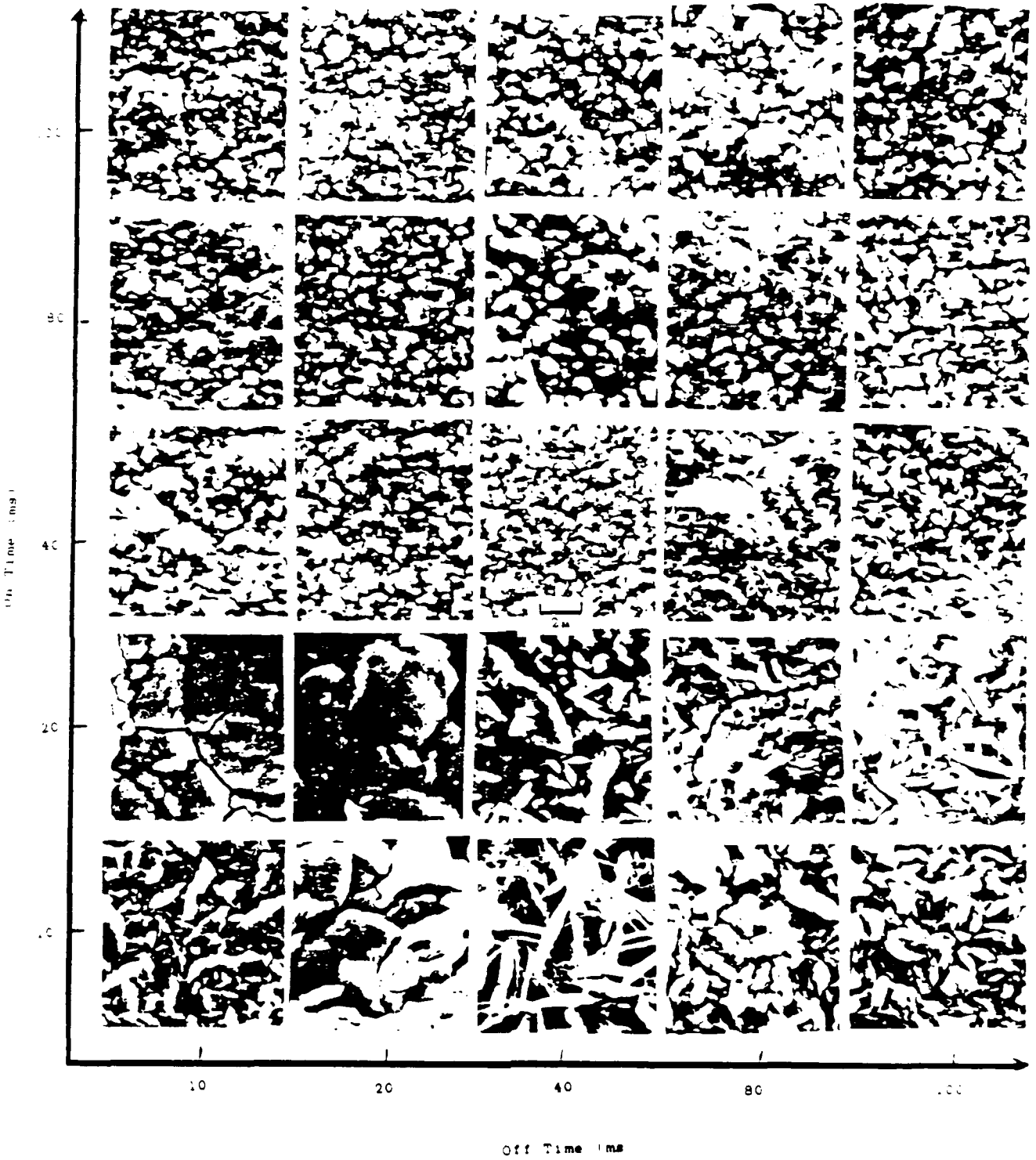

Figure 9. Topographical photomicrographs of LC chromium electrodeposited using low frequency pulse plating.

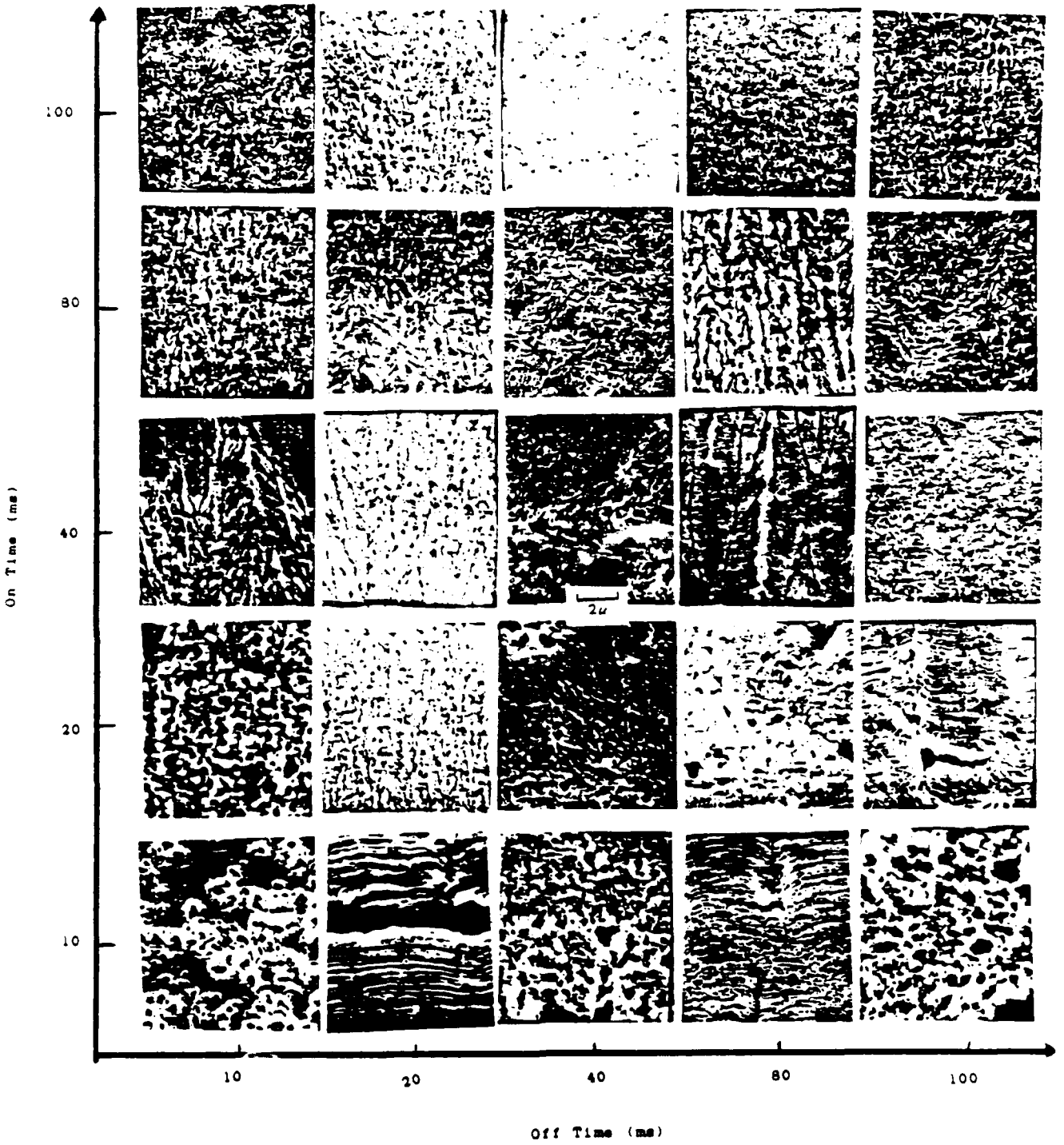

Figure 10. SEM photomicrographs of the cross-sectional microstructure of LC chromium electrodeposited using low frequency pulse plating.

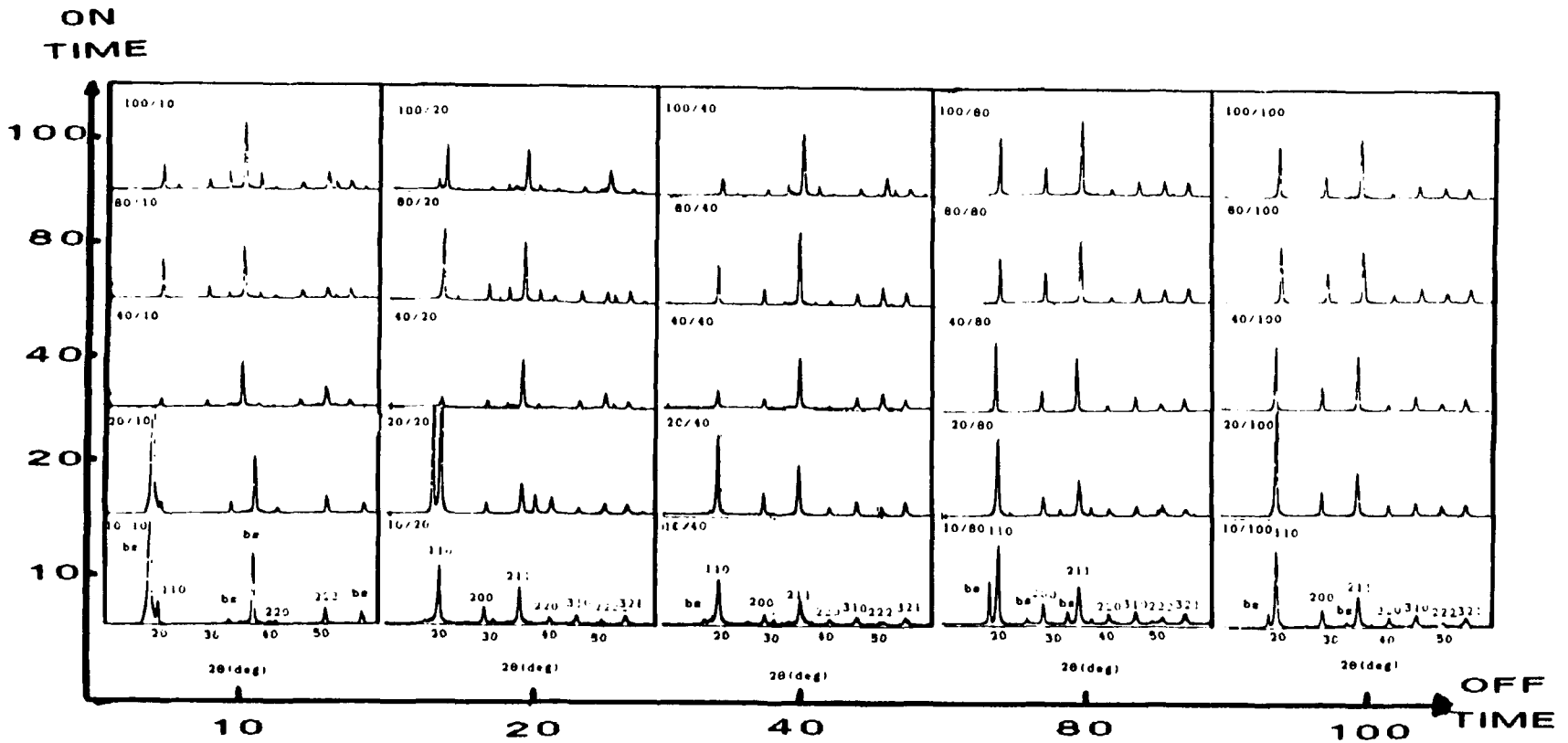

Figure 11. XRD patterns of LC chromium electrodeposited using low frequency pulse plating.

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